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**(54) Method for Producing Expanded Poly(vinyl chloride) materials**

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### **Detailed Description of the Invention**

The present invention relates to a method for producing expanded poly(vinyl chloride) materials. The method for producing expanded poly(vinyl chloride) materials is characterized by the fact that a composition comprising a poly(vinyl chloride), a crosslinking agent and a blowing agent is molded without decomposition of the two agents, then only the surface layer of the molded material is heated intensely to form a crosslinked shell on the surface layer, followed by heating the entire molded material to a temperature higher than the decomposition temperatures of both the crosslinking and blowing agents to effect crosslinking and expansion.

Traditionally, expanded poly(vinyl chloride) materials have been produced by simply heating a poly(vinyl chloride) resin containing a blowing agent to produce expansion, or heating a mixture of resin and blowing agent in a mold to decompose the blowing agent then, after cooling and removal from the mold, heating again to produce a second expansion. Expanded poly(vinyl chloride) materials thus produced are nonflammable and have excellent strength. However, their disadvantage is that they shrink in time.

In order to prevent the shrinkage, the present invention is to provide a method by which crosslinking of poly(vinyl chloride) is achieved to impart appropriate viscoelasticity for expansion then the blowing agent is decomposed to produce expansion. Expanded materials produced by this method have excellent size stability with little shrinkage. Furthermore, by the present invention, in which a poly(vinyl chloride) resin composition containing a crosslinking agent and a blowing agent is molded without decomposition of the two agents, then heating intensely only the surface layer of the molded material to form a crosslinked shell on the surface layer, followed by heating the entire molded material to a temperature higher than the decomposition temperature of both the crosslinking and blowing agents, to effect crosslinking and expansion, expanded materials that do not adhere to the mold, etc. and have a smooth surface and homogeneous microbubbles can be obtained easily.

Because the crosslinked shell formed by the present invention has a high thermal deformation temperature, the molded material does not deform even when heated and its adhesion can be prevented.

The poly(vinyl chloride) resin used in the present invention can be polymers of vinyl chloride, copolymers of vinyl chloride with vinyl acetate, vinylidene chloride, etc., their mixtures with various compatible polymers, plasticizers, fillers, etc.

The crosslinking agent used in the present invention is a compound, which, when heated with the poly(vinyl chloride) resin, can crosslink the resin, such as sulfone azide, azide formate, aromatic azides, etc.

The blowing agent used in the present invention can be azodicarbonamide, dinitroso-pentamethylenetetramine, p,p'-oxybisbenzenesulfonyl hydrazide, etc. Generally, blowing agents with a decomposition temperature about 20°C higher than that of the crosslinking agent are preferable. Also, instead of such a blowing agent, sometimes a solvent with an appropriate boiling point can be used.

For forming the crosslinked shell in the present invention, the surface of the molded material is heated intensely with an infrared lamp, hot wind, electrical heat, etc. to convert a portion or the entirety of the surface into a crosslinked shell. To form the crosslinked shell only on the surface layer without deforming the molded material, it is necessary to heat the surface layer at a high temperature over a short time period while maintaining the molded material at a temperature as low as possible. Generally, the surface is heated to a temperature of 150-200°C.

In the present invention, in order to foam the molded material on which the crosslinked shell has been formed, sometimes it can be simply foamed and expanded in a normal mold or in a mold that can be sealed but is not airtight. Or, it can be foamed in a mold under a higher pressure then the pressure reduced for expansion. Also, it can be foamed in a mold, and then, after cooling and removal from the mold, heated again for a second expansion. Furthermore, it also can be heated in a high pressure container under a higher gas pressure to decompose the blowing and crosslinking agents for partial expansion, and then, after cooling while maintaining the pressure, heating again for the second expansion. Besides, a foamed sheet can be obtained continuously by supplying the mixture from one side of the heating furnace under atmospheric pressure and removing the product from the other side.

The expansion of the composition in the present invention is dependent on composition, temperature, expansion rate, etc. Generally, it is performed so that the resultant elasticity is  $10^5$ - $10^7$  dyne/cm<sup>2</sup>, preferably about  $10^6$  dyne/cm<sup>2</sup>. Under such conditions, expanded materials with homogeneous microbubbles can be obtained efficiently.

In the following, practical examples of the present invention are described.

### Practical Example 1

One hundred parts (by weight) of poly(vinyl chloride), 80 parts (by weight) of dioctyl phthalate, 10 parts (by weight) of azodicarbonamide, 3 parts (by weight) of basic lead stearate and 0.5 part (by weight) of 1,10-decanebissulfone azide were kneaded homogeneously with a roll, then molded by pressing into a sheet of 100 x 100 x 10 mm. The sheet was exposed to infrared radiation at room temperature to heat the entire surface, thereby forming a crosslinked shell. Then it was placed in a mold with an internal size of 200 x 200 x 20 mm and heated at 180°C for 50 minutes for expansion. The resultant expanded material had a density of 0.17 g/cc with a strong and elastic surface layer with a smooth and beautiful surface, and it did not shrink in time. Because the crosslinked shell was formed in advance, it did not form fins due to the seam of the mold, and did not adhere to the mold either.

### Practical Example 2

One hundred parts (by weight) of poly(vinyl chloride), 20 parts (by weight) of dioctyl phthalate, 10 parts (by weight) of isopropanol, 3 parts (by weight) of zinc oxide, 20 parts (by weight) of p,p'-oxybisbenzenesulfonyl hydrazide and 0.5 part (by weight) of 1,10-decanebisazide formate were kneaded homogeneously with a roll, and then molded by a press as in Practical Example 1. The sheet was exposed to 200°C hot wind at room temperature for 5 seconds to form a crosslinked shell on one side of the molded material. The molded material was placed in an autoclave with the side having the crosslinked shell on the bottom, and heated at 170°C for 1 hour under a pressure of 10 atmospheres of nitrogen gas to decompose the crosslinking and blowing agents. The material was cooled to room temperature with high pressure water while the pressure was maintained at 10 atmospheres. The molded material thus obtained was partially expanded to a size of 170 x 170 x 16 mm.

After heating and expansion in an air oven at 140°C for 30 minutes, a molded material was obtained with a density of 0.05 g/cc and homogeneous microbubbles; it had a shape roughly similar to that before expansion.

### Practical Example 3

The same composition as used in Practical Example 1 was press-stretched with a calender to a 1.5 mm thick sheet. The sheet was heated with 200°C hot wind for 3 minutes to expand it in a hot wind furnace with an 80 mesh metal net conveyer. In the furnace the hot wind was blown onto the sheet from both sides of the sheet. The flow rate of the hot wind blown onto the bottom side was higher than that onto the top side in order to obtain even heating of both sides of the sheet.

The resultant expanded sheet had a strong surface and a density of 0.2 g/cc.

## **Patent Claims**

1. Method for producing expanded poly(vinyl chloride) materials, characterized by the fact that poly(vinyl chloride), a crosslinking agent and a blowing agent are kneaded homogeneously and molded without decomposing the two agents, then only the surface of the molded material is heated intensely to form a crosslinked shell, followed by heating the entire molded material at a temperature higher than the decomposition temperature of both the crosslinking and blowing agents for crosslinking and expansion.